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Characterisation of hydrocolloids by vibrational spectroscopy and multivariate analysis

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Abstract

In this research, we proposed an analytical framework data treatment based on dimensionality reduction techniques accompanied by supervised pattern recognition techniques for the analysis of spectral fingerprints of food additives mixtures. Specifically, the objective of this work is to distinguish and classify admixtures of carrageenan, gums and excipients using spectroscopic data. Results demonstrate that this procedure allowed the qualitative discrimination between hydrocolloids blends with a high correct classification rate using a combination of laboratory in-house binary gum admixtures and simulated admixtures.

Keywords: natural gums, gums identification, tara, guar, locust bean, carrageenan, excipient, chemometrics, dimensionality reduction, CLPP

Introduction

Hydrocolloids such as guar gum, locust bean gum (LBG) and tara have the ability to form viscous dispersions and/or gels when dispersed in water. They are used extensively in food industry as thickening agents because they can essentially modify the liquidity of the foods that are added[1]. These hydrocolloids have very similar molecular structure which prevents their identification and even more their quantification in an admixture of natural gums. Existing wet chemistry/ optical techniques currently used for are either insufficient, resource intensive or laborious. Vibrational spectroscopy as Fourier transform mid infrared (FTIR) and near infrared (NIR) are usually used for QA/QC applications in the industry but they are often problematic when the analytes have such similar spectral characteristics.

Materials and methods

Sample

Calibration dataset initially included twenty-nine samples (8 pure (Kappa, Iota, Semiref, Tara, Guar, LBG, KCL and Maltodext) and 21 admixtures in some limited concentration grades). For the creation of a robust and generalized classification model, 1044 simulated samples were artificially generated for covering all the potential combinations using a data augmentation scheme (This work is under review). Figure 1 shows the CLPP space for the training NIR data with the introduction of the simulated spectra. Moreover, an independent dataset including eighty in-house admixtures has also been prepared for validation of the proposed methodology.

Spectral acquisition

The NIR spectrometer model Foss XDS was used for the spectral acquisition of training samples. The NIR spectra were measured in the wavelength region of 400 nm to 2498 nm with 2 nm intervals resulting in 1050 variables. For each sample two ring cups have been filled with the sample and measured in order to obtain two replicates per sample. The final sample spectrum was the average of the two replicates. The acquisition of the testing samples was done with a FOSS DS2500 instrument which has a different spectral resolution than the XDS instrument.

Data analysis

Data analysis was performed with Matlab software (The MathWorks Inc., USA). First, spectral pretreatments were applied. In NIR spectra, visible part (400-1098nm) was cut and then Standard Normal Variate (SNV), detrend and S-Golay filter [polynomial order = 2, frame size = 9] were applied for removing the scatter, linear trends and smoothing the data points respectively. Linear interpolation was applied to the test spectra in order to get the desirable number of variables. Then partial least squares regression (PLSR) coupled with continuous locality preserving projections (CLPP[2]) have been used to tackle this characterization problem. Validation was performed by an independent test set. Training samples were grouped in 5 different classes. The explanation of the model classes is indicated in Table 1. For each training sample, three PLSR reference values

were added corresponding to the carrageenan total, gum total and excipient total respectively. Figure 1 shows the CLPP space for NIR training data. The performance of the proposed framework is shown in Table 2.

Table 1. Definition of the model classes

Class	Description
Pure Carrageenan	Pure Kappa, Pure Iota, Pure Semiref, Kappa + Iota, Kappa + Semiref, Iota + Semiref admixtures
Pure Gum	Pure Tara, Pure Guar, Pure LBG
Pure Excipient	Pure KCl, Pure Maltodext
Carrageenan + Gum admixtures	Admixtures in all potential combination between carrageenan and gums
Carrageenan + Gum + Excipients admixtures	Admixtures in all potential combination between carrageenan, gums and excipients

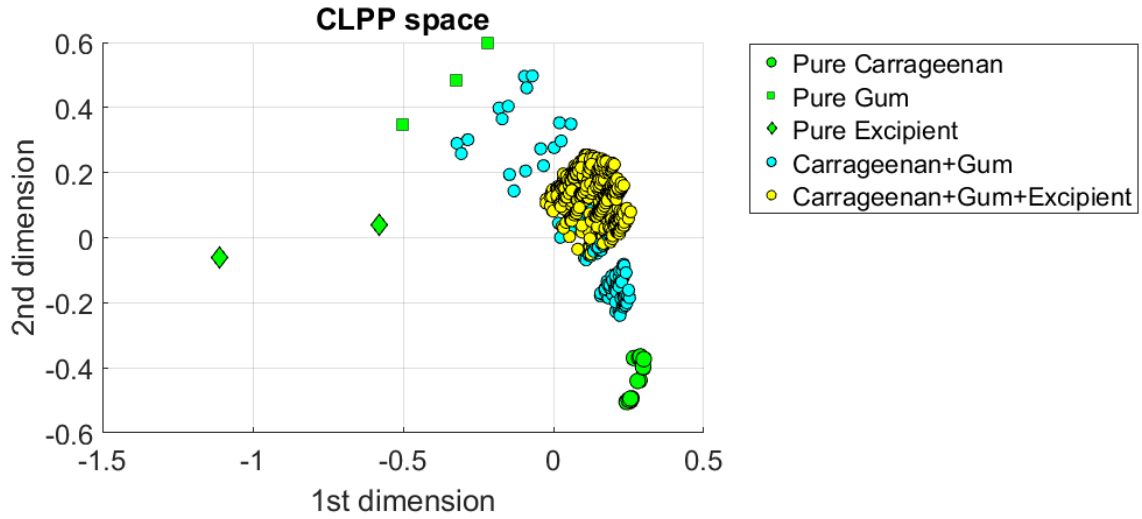

Figure 1. CLPP space for the training NIR spectra

Table 2. Classification and PLS regression results

	Classification technique (CLPP+PLSR)
Classification rate (%) (TP/Total of testing samples)	**100.00 (80/80)**
RMSEP_Carrageenan	**0.056**
RMSEP_Gum	**0.061**
RMSEP_Excipient	**0.058**

RMSEP: root mean square error of prediction

Results and discussion

Results showed that CLPP+PLSR techniques based on NIR data allowed the discrimination between food additives blends with a 100% of correct classification. The same scheme enabled a quantitative determination of carrageenan, gums and excipients with root mean square error of prediction (RMSEP) between 0.056 and 0.061 which indicates that the average difference between predicted and measured response values was very low.

Conclusion

The results confirmed that this method could become an interesting screening tool to quickly categorise the hydrocolloids admixtures. Future work will look at the application of the same framework to identify and quantify each individual member of carrageenan, gum and excipient classes which makes the analytical problem more challenging and complicated.

References

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